ARK:jsg110399/6711002CIP-3.PAMD

In addition, Applicant encloses herewith a copy of PTO-1449 and 892 and copies of the two publications referred to in Applicant's parent application filed on March 1, 1999 as requested by Examiner Popovics.

All fees in connection with this matter should be charged to Deposit Account No. 23-0510.

Respectfully submitted,

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Form PTO-1449 (REV. 8-83)

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ATTY. DOCKET NO. 671.1.002 CIP

SERIAL NO. 09/259,726

APPLICANT(S) William J. Schmidt

FILING DATE March 1, 1999

GROUP 1723

U.S. PATENT DOCUMENTS

Examiner Initial		DOCUMENT NUMBER	DATE	NAME	CLASS	SUBCLASS	FILING DATE IF APPROPRIATE
		5,074,102	Dec. 24, 1991	Wallington M. Simpson et al.			
		5,288,408	Feb. 22, 1994	William J. Schmidt et al.			

FOREIGN PATENT DOCUMENTS

		DOCUMENT NUMBER	DATE	COUNTRY	CLASS	SUBCLASS	TRANSLATION YES	TRANSLATION NO

OTHER DOCUMENTS (Including Author, Title, Date, Pertinent Pages, Etc.)

		Ultra Filtration Handbook, Munir Cheryan, Technomic Publishing Co., pp. 276-277 (1986)
		GEA Westfalia Separator AG "Separators and Decanters for Gelatine Production"

Examiner

DATE CONSIDERED.

* EXAMINER: Initial if citation considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

Notice of References Cited

Application No.	Applicant(s)		
09/259,726	Schmidt		
Examiner	**Group Art Unit**	**Page** 1 **of** 1	
Popovics	1723		

U.S. PATENT DOCUMENTS

*		DOCUMENT NO.	DATE	NAME	CLASS	SUBCLASS
*	A	5,945,001	8/99	Schmidt	210	773
	B					
	C					
	D					
	E					
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	G					
	H					
	I					
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Part of Paper No. 7

Munir Cheryan, Ph.D.

ULTRAFILTRATION HANDBOOK

TECHNOMIC PUBLISHING CO., INC.
LANCASTER · BASEL

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ISBN No. 87762-456-9

Table 1.1. Characteristics of ideal membrane processes.

Process	Driving Force	Permeate	Retentate*
Osmosis	Chemical Potential	Water	Solutes
Dialysis	Concentration Difference	Water + Small Molecules	Large Molecules
Ultrafiltration	Pressure	Water + Small Molecules	Large Molecules
Reverse Osmosis	Pressure	Water	Solutes
Electrodialysis	E.M.F.	Water + Ionic Solutes	Nonionic Solutes
Microfiltration	Pressure	Water + Dissolved Solutes	Large Suspended Particles

*Also includes water

molecules or fine colloidal suspensions. Microfiltration is also a method for essentially separating suspended particles from dissolved substances in a feed stream, provided the particles meet the size requirements for microfiltration membranes. Figure 1.2 shows some typical examples of components that fall under these three processes. It is customary to refer to "molecular weight cut-off" instead of particle size per se when attempting to classify membranes within ultrafiltration itself; thus ultrafiltration (or UF as it is commonly referred to) covers "particles" or molecules that range from about 1000 in molecular weight to about 1,000,000.

Ultrafiltration, together with its sister process, hyperfiltration or reverse osmosis (RO), thus constitute the first continuous molecular separation processes that do not involve a phase change or interphase mass transfer and this is perhaps what excites workers in the area of food, pharmaceutical and biological processing. In its simplest form, as shown in Figure 1.3, RO and UF consist merely of pumping the feed solution under pressure over the surface of a suitably supported membrane, of the appropriate chemical nature and in the optimum physical configuration. In the UF process, the pressure gradient across the membrane would force solvent and smaller species through the pores of the membrane, while the larger molecules would be retained. The retained phase, or "retentate" or "concentrate" stream as it is referred to, will thus be enriched in the retained macromolecules, while the permeate stream will be depl ted of the macromolecules. The retentate will of course contain some of the permeable solutes also. In fact it may be the very same or higher concentration than in the permeate stream, depending on that component's rejection by the membran . However, since th

Figure 1.1 shows a classification of various separation processes based on particle or molecular size and the primary factor affecting the separation process. The five major membrane separation processes, reverse osmosis, ultrafiltration, microfiltration, dialysis and electrodialysis, cover a wide range of particle sizes, matched in versatility only by centrifugal processes. However, an absolute requirement for centrifugal processes is the existence of a suitable density difference between the two phases that are to be separated, in addition to the two phases being immiscible. Membrane separation processes have no such requirement, and indeed, the real value of ultrafiltration and reverse osmosis is that they permit separation of dissolved molecules down to the ionic range, provided the appropriate membrane is used.

Among membrane separation processes itself, the distinction between the various processes is somewhat arbitrary and has evolved with usage and convention. Table 1.1 shows the characteristics of various membrane processes. Osmosis (to be discussed in detail in Section 1.C.), is the transport of solvent through a semi-permeable membrane from the dilute solution side to the concentrated solution side of the membrane. It is driven by chemical potential differences between the water on either side of the membrane. With an ideal semi-permeable membrane, only water should permeate through the membrane. The common laboratory technique of dialysis, on the other hand, is primarily a technique for purifying macromolecules, such as desalting of proteins, and the primary driving force would be difference in concentration of the permeable species between the solution in the dialysis bag and on the outside of the bag. Electrodialysis relies primarily on voltage or electromotive force and ion-selective membranes to effect a separation between charged ionic species.

What distinguishes the more common membrane processes—microfiltration, ultrafiltration, and hyperfiltration (reverse osmosis)—is the application of hydraulic pressure to speed up the transport processes. However, the nature of the membrane itself controls which component permeates and which component is retained. In its ideal definition, reverse osmosis or hyperfiltration retains *all* components other than the solvent (water) itself, while ultrafiltration retains only macromolecules or particles larger than about 10–200 Å (about 0.001–0.02 μm). Microfiltration processes, on the other hand, are designed to retain particles in the "micron" range, that is, suspended particles in the range of 0.10 μm to about 10 μm. In conventional usage, particles larger than 10 μm are best handled by conventional filtration processes. Thus, in its broadest sense, reverse osmosis or hyperfiltration is essentially considered to be a dewatering technique, while ultrafiltration can be looked at as a method for simultaneously purifying, concentrating, and fractionating macro-

Table 8.25. Ultrafiltration of animal processing industry waste stream using Kalle PA-40 ultrafiltration membrane (Source: Beer, 1979).

Component	Feed (gm/L)	Retentate (mg/L)	Permeate (mg/L)	Percent Reduction
COD	14,514	78,000	3,677	75
Fat	3,700	14,800	84	98
Protein	3,100	15,500	2,000	36

treatment or surcharges. The retentate can be sold as "brown grease," or incorporated into animal feed, fertilizer or as a soil conditioner.

Gelatin is another animal processing industry product that can be processed by ultrafiltration. Gelatin is not exactly a waste product, although it is obtained from animal by-products that are not ordinarily consumed. Gelatin is widely used as a glue, in pharmaceutical preparations, photographic products, and, of course, in its edible form, as a popular dessert. It is manufactured by aqueous extraction at high temperatures, using either acid or alkali, from the skins, hides, and bones of animals. The extract is typically dilute, about 2–5% protein, and contains high quantities of ash. This gelatin extract must be de-ashed (usually by ion-exchange) and concentrated (by evaporation) to yield a product of about 90% protein, less than 0.3% ash and no more than 10% moisture.

Ultrafiltration has a number of specific applications in the gelatin industry: (1) Preconcentration of the dilute extract solution prior to evaporation, (2) simultaneous reduction in ash components, thus upgrading the product, and (3) reduction in lower molecular weight components to improve the gelling properties of the product. In addition, the overall yields can be increased. This can be done by increasing the number of extraction stages. The extracts from the additional stages will have a lower solids content, which would normally be uneconomical to process. The C_G values of gelatin are 20–30% protein, which limits practical ultrafiltration to about 18–20% protein in the retentate (Figure

Table 8.26. Analysis of water samples from ultrafiltration of poultry wastewater (Shih and Kozink, 1979).

Component[a]	Wastewater (mg/liter)	Concentrate (mg/liter)	Permeate (mg/liter)	% Total reduction
TS	1278	5398	240	85
COD	1968	9116	131	95
Ash	104	276	48	63
TKN	82	372	14	86
Protein	492	2013	37	94

[a]TS: total solids, COD: chemical oxygen demand, TKN: total Kheldahl nitrogen.

8.27). This usually implies a 50–80% reduction in volume. However, owing to the large increases in viscosity, it may not be practical to go very much above 10% protein (Poulsen, 1983).

Flux with DDS plate units averaged 4–18 LMH for the GR8P membrane and 10–60 LMH for the GR6P UF membrane over the range of 3–20% gelatin concentration (Alred, et al., 1980). Donnan effects play an important role in the ultrafiltration of gelatin. Negative rejections of calcium occurred below pH 4, and sometimes calcium rejections of up to −380% were observed (Figure 8.28). Most negative rejections were obtained at low pH and high concentrations of gelatin. The Donnan-enhanced transport was increasingly counter-balanced as the membrane became more gel-polarised (e.g., at higher transmembrane pressure and/or higher gelatin concentrations). pH values above 4 tended to strongly reject the calcium, due possibly to the binding of the calcium to the protein, which has a net negative charge above its isoelectric point of 4.5.

Flux with Abcor spiral-wound modules is 50–80 LMH at 2–3% gelatin concentration, transmembrane pressure of 50 psi, a pressure drop of 20 psi per module and a temperature of 50C. It drops to about 3–4 LMH at 18% gelatin concentration. An economic analysis of the process by Abcor, based on a 20,000 kg/hr plant feed capacity, indicates a potential

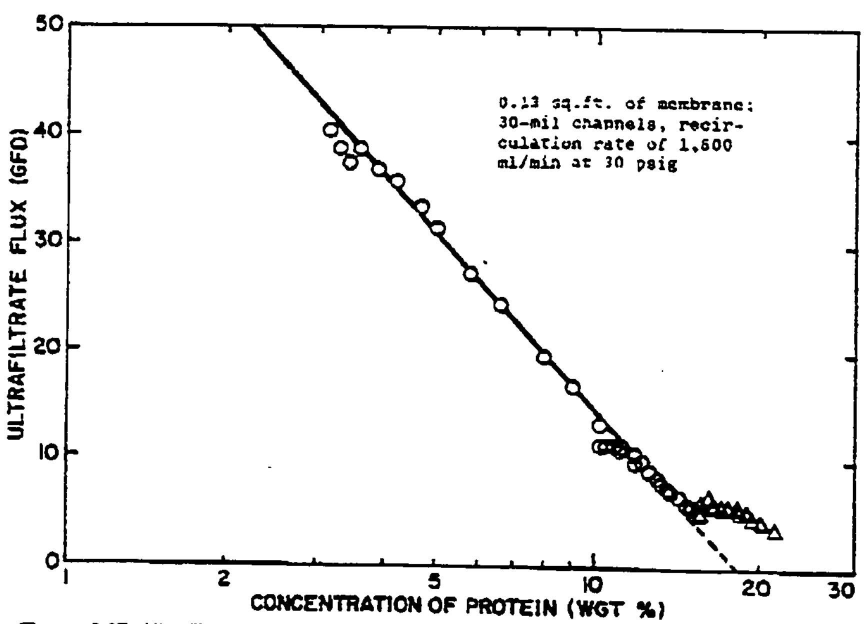
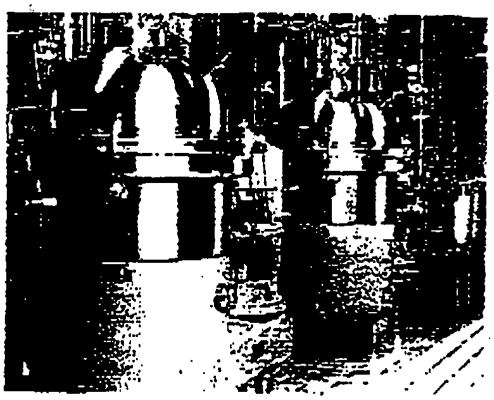
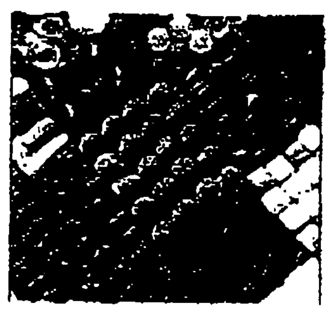


Figure 8.27. Ultrafiltration of gelatin: relationship between gelatin concentration and flux (Source: Porter and Michaels, 1971).

Separators and Decanters for Gelatine - Production





GEA

Process Technology
Division

Separators and decanters for gelatine production

J. Schwennen

List of contents

1. State-of-the-art of gelatin production

The term „gelatine" was first encountered around 1700, and is derived from the Latin word „gelatus", namely „frozen" or „rigid". The first report of technology used for edible gelatine production goes back to 1685, although it was 1870 before it was discovered that gelatine is a protein.
Today, more than 160,000 tonnes of gelatine are produced throughout the world every year. Of this figure, approx. 65% is used in the food industry, 20% in the pharmaceutical industry and 15% in the photographic industry.

Separators and decanters are used in the process of manufacturing gelatine; they meet the users' needs in respect of continuous operation and greater efficiency. Installations using centrifugal separation technology perform their tasks more simply in comparison with filter installations.

This prospectus details the various process stages in gelatine production. In particular, the prospectus explains the use of centrifugal separators for solving various problems of separation.

1.1 Fields of application for gelatine

After having been dried, gelatine is sold as sheet gelatine or in various grain sizes, whereby the nature of the gelatine is particularly important for the customer.
Most gelatine is used in the food industry:

- — Confectionery
- — Beverage industry
- — Dairy industry
- — Baking products
- — Meat industry

The pharmaceutical industry is a second area of application for gelatine:

- — Capsules
- — Tablets
- — Coated tablets
- — Gelatine sponge
- — Blood substitutes
- — Dietetics
- — Cosmetics

Gelatine is also used in the photo industry, where it is known as technical gelatine. The difference between technical gelatine and edible or pharmaceutical gelatine is that the food technology aspects of the gelatine are not essential to the same extent. The other parameters such as Bloom coefficient, viscosity, colour, clarity, pH value, etc. are applicable for both fields of production. This is one reason for many manufacturers of technical gelatine to make no distinction, as it can be manufactured from type A gelatine as well as from type B gelatine.

This gelatine is used for the production of:

- — Film products
- — Paper
- — Printed products
- — Laboratory articles

1.2 Compositi n of g latine

Gelatine is animal protein in very pure form, and is thus a product with a very natural origin. The basic substance required for producing gelatine is collagen. Collagen is the most common protein in animals as well as in humans (around 30%). Collagen is found in particularly high concentrations in gelatine raw material such as bones, skin and connective tissue.

As is the case with any protein, the basic modules of collagen are amino acids. A chemical analysis indicates that gelatine consists of approx. 84-90% protein and 1-2% mineral salts. The remainder is water. It does not contain any preservatives or other additives, and is free of cholesterol and purines.

The fact that gelatine protein is made up of various amino acids is particularly important in terms of nutritional physiology. Of the ten essential amino acids which the human organism is not able to form itself, edible gelatine alone contains nine; they represent approx. 23% of the total gelatine-protein content.

Fig. Illustration of average values of amino acids

Amino acid composition of gelatine	Average value of amino acids in %
Alanini	11.0
Arginine	9.0
Asparaginic acid	6.7
Cystein	0
Cystin	0.1
Glutamin acid	11.4
Glycin	27.0
Hystidin*	0.8
Proline	16.0
Hydroxyproline	13.8
Isoleucine*	1.5
Leucine*	3.3
Lysine*	4.3
Methionine*	0.8
Phenylalasine*	2.4
Serine	4.1
Threonine*	2.2
Tryptophane*	0
Tyrosine	0.3
Valine*	2.7

* Essential amino acid

The fact that tryptophane is the only essential amino acid which is almost completely absent in edible gelatine means that the biological value of edible gelatine is low. However, it becomes a full-value protein together with other protein media. In consequence, it is not regarded as a food additive, it is regarded as an independant foodstuff.

2 Raw materials

Selected bones, calf and cattle skins and pig skin strips are used as the main raw material for gelatine production. Experience has shown that between 6 and 8 kg raw material are necessary for the production of 1 kg gelatine.

Pig skin strips

are obtained either fresh and cooled or deep-frozen from meat processors, and are stored in the same state in a refrigerated facility until they are processed.

Calf and cattle skins

are cleaned in the tanneries and are split after alkaline pre-treatment. The skin side is used for leather, whereas the underside, which is virtually pure collagen, is excellent for the production of gelatine. This split is preserved with salt and quick lime and stored until it is processed.

Fresh bones

are cooled and sent in this state by the shortest route from deboning halls and meat processors to specially designed defatting installations, where they are processed immediately. The bones are broken down gently, washed and defatted with hot water, whereby the temperature of the water does not exceed 70 °C during the fat extraction process. Before they are dried, the bone pieces are sorted into different grain sizes, and they are then dried and stored. The subsequent treatment of this raw material over several days with diluted hydrochloric acid at low temperatures (maceration) removes phosphate mineral.
After neutralisation, the demineralised bones are available as so-called ossein for gelatine production. The di-calcium phosphate which is obtained as a by-product is a valuable fodder additive.

3 Processes for gelatine production

There are two basic types of pre-treatment used in the production of gelatine. On the one hand, the alkalin method is used for type B gelatine (basic). On the other hand, the acid procedure is used for type A gelatine (acid).

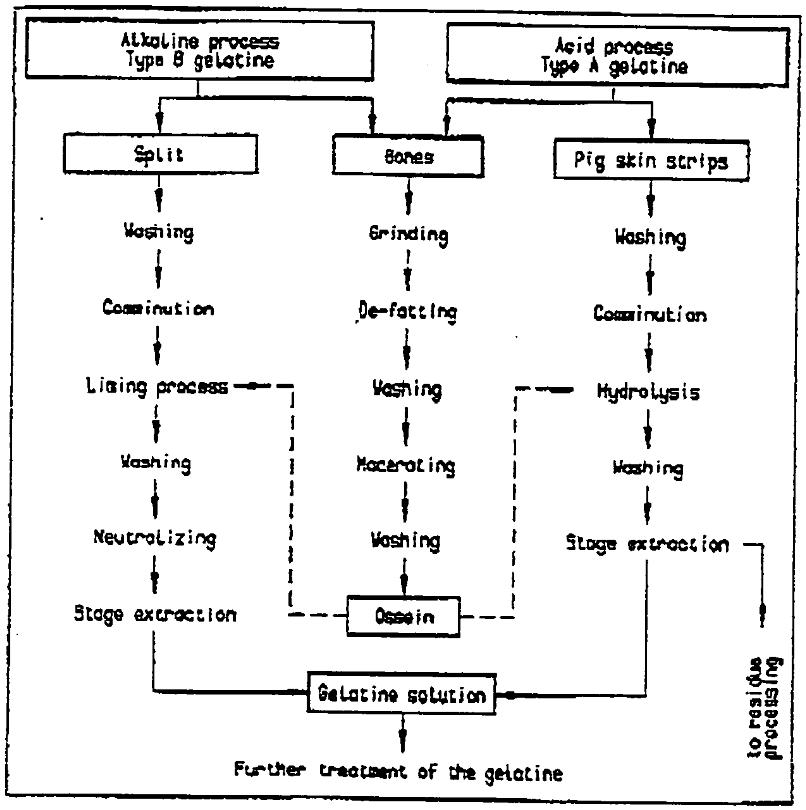


Fig. Flow sheet illustrating gelatine production

3.1 Type B gelatine, alkaline pr cess

In order to remove preserving salt, cattle and calf split is washed, broken down and treated by the so-called liming process. The effect of lime hydrate over 2-4 months partially breaks down the polypeptide chains into smaller fractions. In addition, the existing cross links between the chain molecules in the collagen protein are also partially broken down, whereas all non-proteins and associated proteins are removed.
The pre-treated material is washed and neutralised, and the salts which have occurred are washed out. The product is then ready for the extraction process.
As the water is replaced more than 20 times during the pre-treatment of the raw material, approx. 1000 l water are required for 1 kg gelatine.
The process described above is also applicable for ossein. However, the alkaline process takes only 1 - 2 months as the maceration process has already acted on the polypeptide chains.
However, acid treatment can also be used for the ossein.

3.2 Typ A g latin , acid treatment

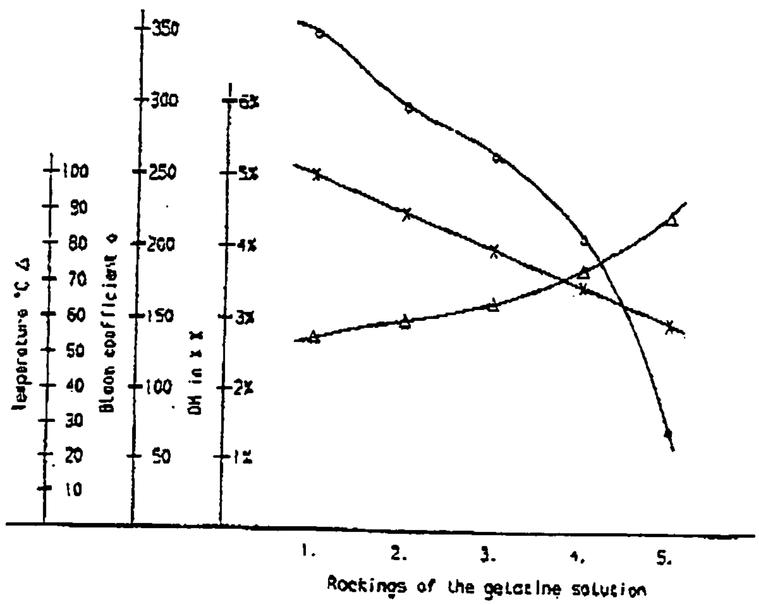
This is currently the most common method, and is suitable primarily for pig skin strips; because of their relatively low age, pig skin strips are more suitable for extraction in comparison with the time-consuming process used for cattle or calf skins.

The single-stage method is based on macerating and breaking down the raw collagen by the action of strong acid; the broken down pig skin strips are placed in an approx. 3% acid solution for around 24 hours. After the acid treatment, the excess acid is partially neutralised and the salts are also washed out by means of repeated washings with water.

4 Extraction

The materials which have been pre-treated in this way are mixed with hot water and are then treated by means of discontinuous stage extraction. A thermal protein denaturation process is initiated when the materials are heated in an aqueous solution at pH 6.0 - 6.5 (split and ossein); the aim of this process is to break down the hydrogen bond system which is the most important stabilising factor of collagen structure, although the process is not designed to break down the peptide fragments.
The pig skin strips are extracted at pH 4.3 - 4.7, which means that there is a partial overlap between the internal breakdown of collagen and thermal protein denaturisation.
During the extraction process for split and ossein, the thorough precleaning process means that there are hardly any residues. On the other hand, during the extraction process of pig skin strips, residues of up to 30% fat and 2-3% protein are likely depending on the composition of the raw materials.
Starting at 50°C and as the temperature rises, 4 - 5 rackings are taken off in the form of an approx. 5% gelatine solution, whereby the gelatinising power of the gelatine recovered in this way declines in each successive racking as a result of increasing thermo-hydrolysis; in consequence, the individual rackings are processed separately.

The following diagram illustrates that the gelatinising power of the gelatine declines as a result of increasing thermo-hydrolysis. For this reason it is recommended that the individual rackings be processed separately.



5 Gelatine clarificati n

5.1 Pre-clarification and defatting

It is first of all important to remove fibres and fat residues from the raw material from the gelatine solution obtained during the extraction process.

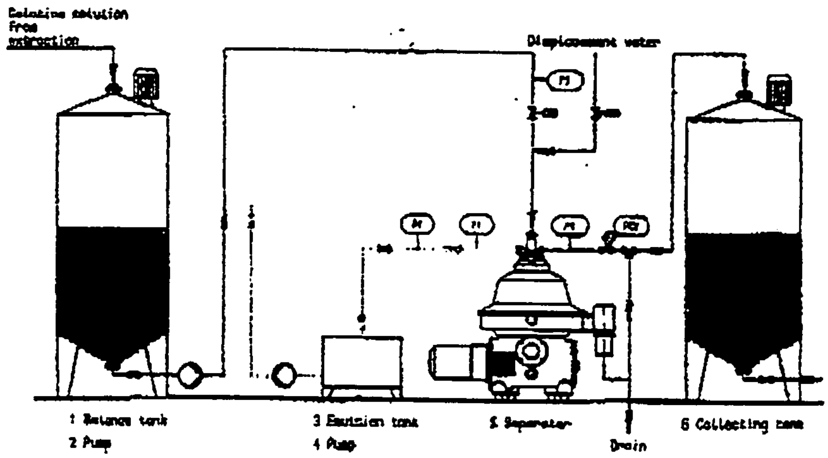


Fig. Installation diagram - gelatine-defatting and clarification

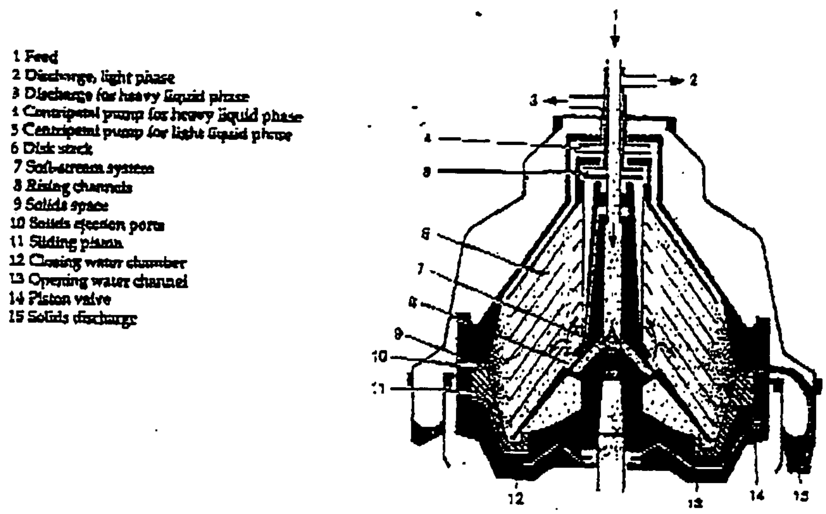
The separating temperature, the DS content and viscosity vary in the different rackings, and must not be changed before centrifugal separation.
If skimming separators are to be used, the gelatine broth must first pass through a screen in order to remove coarse particles which might block the rising channels of the disk stack.

Fig. Types and ratings of self-cleaning separators

Type	Nominal rating up to l/h
MSD 90-01-076	4000 - 5000
MSD 130-01-076	6000 - 7500
MSD 170-01-076	10000 - 12000
MSD 200-01-076	12000 - 14000
MSD 300-01-076	16000 - 18000

The process uses self-cleaning separators such as those encountered in the dairy industry. Separators with a „soft-stream inlet" guarantee gentle product treatment and excellent defatting efficiency. These separators are used if a continuous production process is required. The product is separated in the disk stack into a light and heavy liquid phase, and any solids are discharged into the solids space. The solids space features a double-conical design, and can be opened and closed by the sliding piston which moves axially downwards. During the production process, the bowl can be opened and closed at definable intervals by lowering the sliding piston. The solids which build up are ejected during this process. After the end of production, the separator is automatically cleaned chemically (CIP = cleaning in place). Operation can be fully automated by means of appropriate control units.

Fig. Bowl cross-section of a self-cleaning separator
Type MSD



5.2. Centrifugal polishing

Self-cleaning centrifugal precoat filters in which the diatomaceous earth retains fine impurities have previously been used for centrifugal polishing. Extensive trials have confirmed that high-performance clarifiers of type CRA 160-96-076 considerably exceed the clarification performance of diatomaceous earth filtration and also extend the operating time of downstream layer filtration.

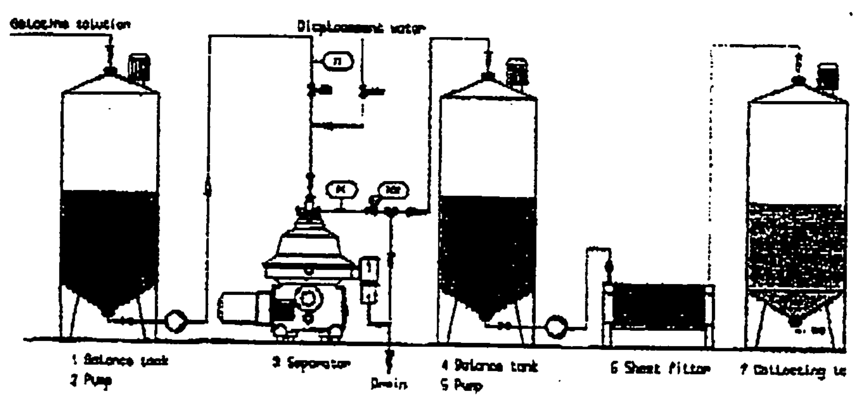


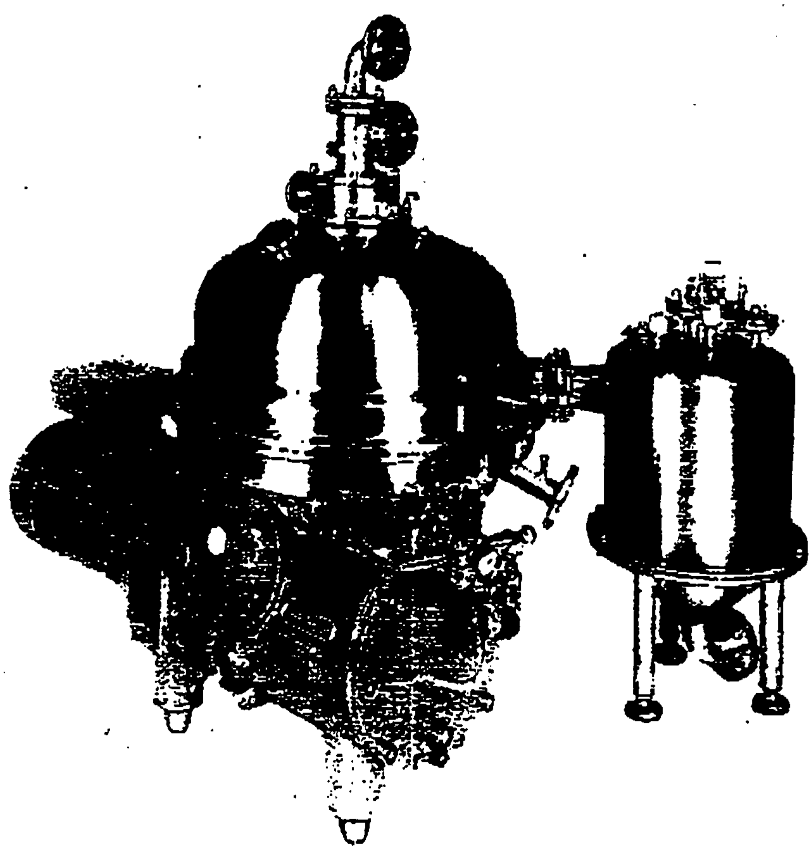
Fig. Installation diagram centrifugal polishing of gelatine

This considerable clarification performance is achieved by centrifugal acceleration equivalent to 15.000 times the earth's gravity and which thus enables very fine trub particles to be removed.

Further advantages of the high-performance clarifier:

- o Constant throughput throughout the entire production process
- o Self-cleaning of bowl and disks by means of partial and total ejections with a hydraulically actuated annular piston
- o No costs incurred for filter aids
- o No additional costs for disposing of spent diatomaceous earth
- o Taste not affected by filter aids
- o Simple and time-saving cleaning by means of „CIP"
- o Low personnel costs

Depending on the type of gelatine to be processed, this type CRA 160-96-076 separator is suitable for centrifugal polishing of 7000-8000 l/h.



5.3 Ion exchangers

Ion exchangers filled with resins or analogue processes represent the next cleaning stage for specific applications of the intended type of gelatine. Depending on the specific requirements, it is possible to remove calcium and sodium ions, acid residues or other salts more or less completely from the gelatine.

5.4 Layer filtration

Sheet filters as used in the beverage industry are used as the final cleaning stage.

6 Concentrati n

Single-stage or multi-stage circulation evaporators are used for concentrating the glue broths from 5% DS to 35% DS. In order to minimise the Bloom loss during processing, the gelatine broth must not be exposed to excessive heat. Colour is also a major factor in assessing the quality of gelatine. The quality of gelatine is determined by its brightness and clarity. As is the case with gelatine consistency, heat during the production process affects the colour and clarity of the end product. It is therefore recommended that the concentration process be performed under vacuum and at a temperature of 50 - 70 °C.

7 Filtering

The highly viscous gelatine solutions are passed through polishing filters consisting of cellulose plates, which remove the remaining very fine suspended particles.

8 Sterilizing

The gelatine solutions are passed through a flash heater, and are then sent to a sterilising station. As the composition of gelatine solutions means that they are sensitive to heat, the temperature of 120 - 145°C necessary for sterilising should be attained as quickly as possible, and the solutions should not be kept at this high temperature for more than 3 - 8 seconds.

9 Drying

The highly concentrated gelatine solutions are cooled via so-called scraped surface heat exchangers, and when they have set they are pressed through a perforated disk. This results in long strands which are spread evenly over the drier belt. This is where the gelatine is dried gently at a low temperature by means of sterile air which has been treated accordingly. The resultant brittle gelatine is then broken and ground to a specific and standard grain size.

10 Comminution, standardising, sorting

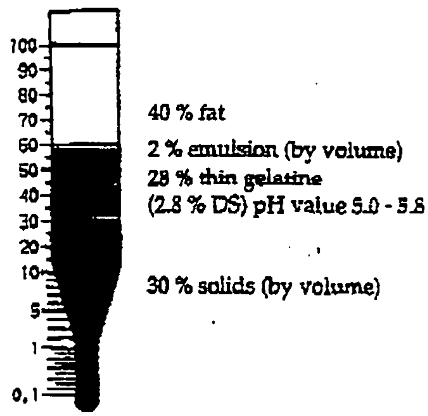
Following a physical, chemical and bacteriological check, the gelatine is released for its definitive applications. In order to satisfy the needs of individual customers, the gelatine can be comminuted, standardised and sorted in order to meet the specific requirements.

11 Special pr cesses

11.1 Processing the residues from extraction

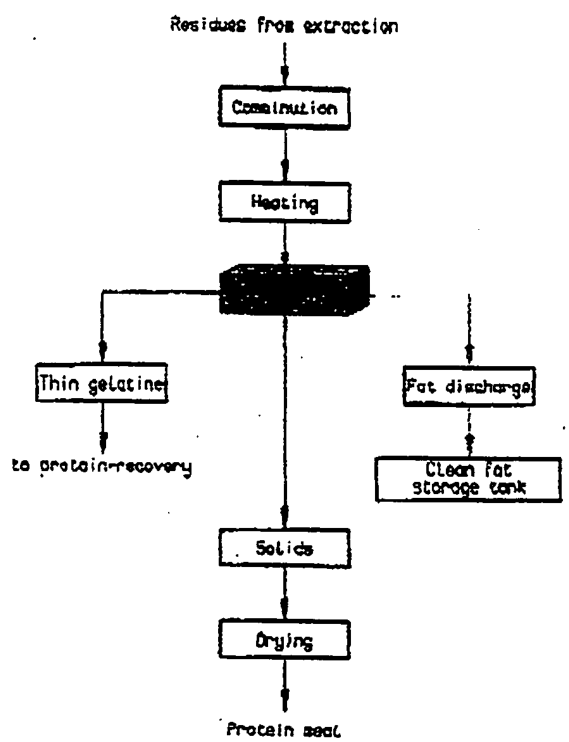
In the extraction process of pig skin strips, there are residues of approx. 15% fat and 2.7% undissolved solids (in relation to the initial product). Together with the thin gelatine, this residue consists of the following (at the point of racking):

Centrifugation sample:



The flow chart illustrates the individual stages of technology involved in recovering the residues, as well as the ecological benefit and the use of the three phases which are obtained. The process can be optimised by breaking down the residues and heating them to the necessary temperature. A decanter is then used for separating the phases into pure fat, thin gelatine and solids. The separated phases are processed accordingly or sent to other process stages.

Fig. Flow sheet for recovering residues



The pure fat which is obtained has an ffa content of <3% and a water content of <0.2%. The fat content in the thin gelatine is <0.3%, and can be recycled back into the extraction process.
The solid component can be dried to meat meal with a protein content of approx. 70% and a fat content of <10% (in relation to DS content).

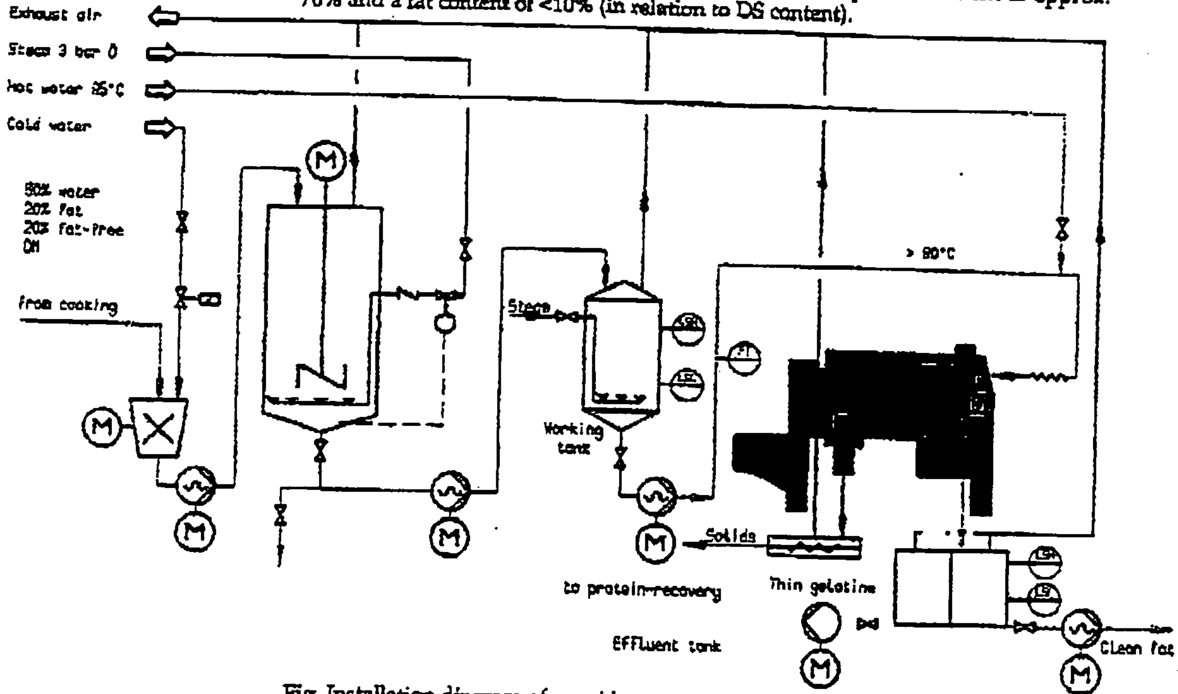
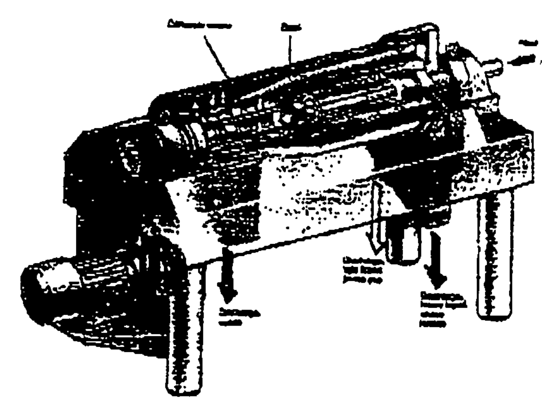


Fig. Installation diagram of a residue processing line

The separating decanter CA 450-04-00 can be used for separating the residue suspensions (or the CB 300-04-00 for lower throughputs).
The light phase which is discharged (fat) does not need to be treated for throughputs up to 5000 l/h (2200 l/h). For higher throughputs of up to 8000 l/h (3500 l/h), it is necessary to subsequently separate the fat phase.

In the gelatine industry, the separating decanter enables the residues to be treated appropriately as the two liquid components - fat and thin gelatine - are discharged separately. The centrifugal force causes the solids in the liquid mixture to settle out on the bowl wall, where they are conveyed continuously to the solids discharge by means of a scroll. The thin gelatine is drawn off from the liquid sump. The fat phase which accumulates in the cylindrical part of the decanter is discharged via the internal ring dam. Both liquid phases are discharged under gravity.

Fig. Separating decanter type CA 450-04-00



11.2 Treatment of effluent

The waste cleaning water which is obtained during gelatine production is treated in a special flotation plant. The flotate is skimmed off and heated to 95 °C in a tank which can be heated with direct steam, and is then treated in the residue processing installation. In this way, approx. 2.0% protein meal (protein content >50%) is obtained in relation to the weight of the input raw material. The purified fat can be used as technical fat (ffa content >15%).

Centrifugation sample of the heated flotates:

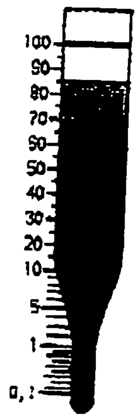
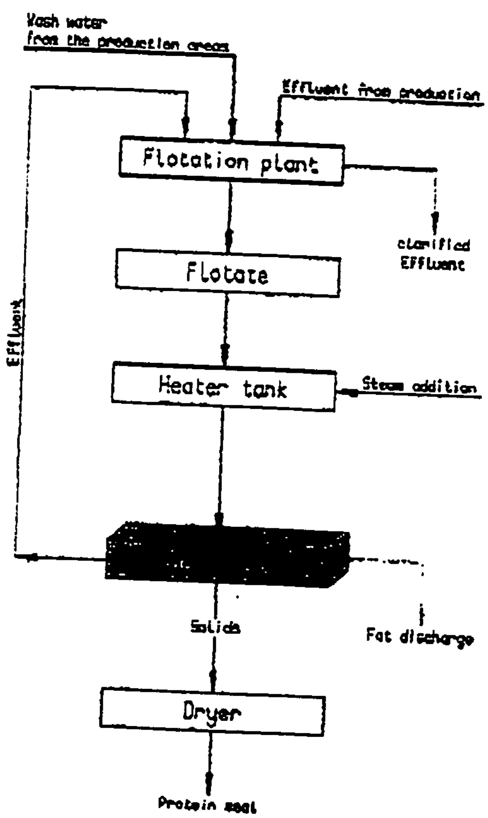
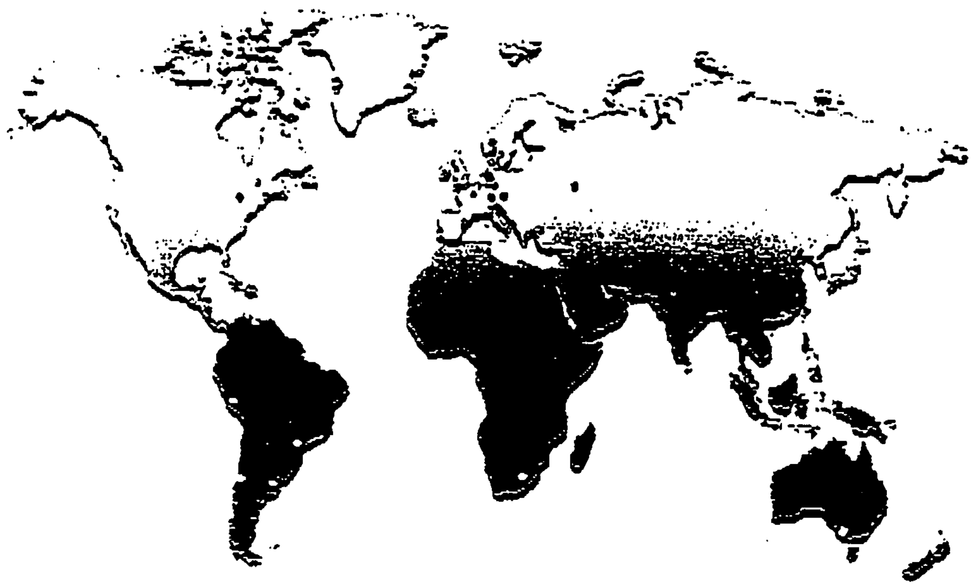


Fig. Processing of the flotate in the effluent cleaning plant (flotation)





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Subject to modification